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मानक

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IS 880 (1956): Tartaric Acid [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

SPECIFICATION FOR TARTARIC ACID

Second Reprint DECEMBER 1996
(Incorporating Amendment No. 1)

UDC 661.733.3

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Indian Standard

SPECIFICATION FOR TARTARIC ACID

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 9 July 1956, on approval by the Chemical Division Council of the draft finalized on 27 April 1956 by the Fine Chemicals (Organic and Inorganic) Sectional Committee.

0.2 Taking into consideration the views of the consumers and technologists, the Sectional Committee responsible for the preparation of this standard felt that it should be related to the trade practices followed in this field in the country. Furthermore, due weightage had to be given to the need for international co-ordination between standards prevailing in different countries of the world. These considerations led the Sectional Committee to draw assistance from the following publications:

BRITISH PHARMACOPOEIA, 1953.

PHARMACOPOEIA OF THE UNITED STATES OF AMERICA, 14 ed. 1950

*ANALAR STANDARDS FOR LABORATORY CHEMICALS. 4th ed. London. The British Drug Houses Ltd., and Hopkin & Williams Ltd. 1949.

0.3 This standard requires reference to the following Indian Standard Specifications:

IS: 264-1950 NITRIC ACID

IS: 265-1950 HYDROCHLORIC ACID

*The information from this publication has been used with the permission of the publishers.

IS: 266-1950 SULPHURIC ACID

IS: 323-1952 RECTIFIED SPIRIT

IS: 460-1953 TEST SIEVES

IS: 695-1955 GLACIAL ACETIC ACID

0.3.1 Wherever a reference to any specification mentioned under 0.3, except IS: 460-1953, appears in this standard, it shall be taken as a reference to the latest version of the specification.

0.4 For particle size, the aperture sizes are based upon Indian Standard Specification for Test Sieves (IS: 460-1953). Where these sieves are not available, other equivalent standard sieves, as judged by the aperture, may be used. For the purpose of comparison, the corresponding sizes according to British Standard Sieves and sieves of the American Society for Testing Materials (ASTM) are given in Appendix C.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1949 Rules for Rounding Off Numerical Values; the number of places retained in the rounded off value should be the same as those of the specified value in the standard.

0.6 This standard is intended chiefly to cover the technical provisions relating to the supply of the material, and it does not include all the necessary provisions of a contract.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for Tartaric Acid of Technical, Pure and Analytical Reagent grades.

2. GRADES

2.1 There shall be three grades of the material, namely (a) Technical, (b) Pure, and (c) Analytical Reagent.

3. SAMPLING

3.1 Representative samples of the material shall be drawn as prescribed in Appendix A.

4. REQUIREMENTS

4.1 Description — The material shall be in the form of colourless or translucent crystals or a white powder. It shall be odourless and strongly acidic in taste. The solution in water shall be dextro-

rotatory. In case of synthetic material there may not be any rotation.

4.2 The material shall comply with the requirements given in Table I, when tested according to the methods prescribed in Appendix B. References to the relevant clauses of Appendix B are given in col 6 of Table I.

5. PACKING AND MARKING

5.1 The material of the technical and pure grades shall be packed as agreed between the purchaser and the vendor.

5.1.1 The analytical reagent grade of the material shall be packed in glass bottles.

5.2 The packages shall be securely closed and marked with the name of the manufacturer; weight and grade of the material in the package; recognized trade mark, if any; and the date and year of manufacture.

5.2.1 The container for analytical reagent grade shall be labelled with the full analytical data of the characteristics prescribed in col 5 of Table I.

AMENDMENT NO. 2 MAY 1996 **TO** **IS 880 : 1956 SPECIFICATION FOR TARTARIC ACID**

(Page 2, *Foreword*, clause 0.1) — Insert the following new clauses after 0.1 and renumber the subsequent clauses:

0.2 Tartaric acid (2, 3-dihydroxysuccinic acid) having empirical formula $C_4H_6O_6$ and molecular mass 150.00 is a byproduct of grape fermentation industry. Commercially it may be synthesized by epoxidation of maleic acid followed by cleavage of epoxide. Its molecular formula is $[CH(OH)COOH]_2$.

0.3 Tartaric acid by virtue of having an asymmetric carbon atom possesses optical isomerism and as expected, is present in four forms. Two of these forms, namely, D (-) tartaric acid and L (+) tartaric acid exhibit optical rotation (each of these differing in signs of rotation) whereas the other two forms, namely, *racemic* and *meso* tartaric acid being 1:1 mixture of the former two-optically active forms, do not exhibit any rotation. The *racemic* variety can be resolved in optical isomers after special treatments whereas *meso* variety cannot be resolved by any method. Synthetic tartaric acid, except when resolved, is obtained in *racemic* form and hence does not have any optical rotation. The natural tartaric acid, depending upon its optical purity will show some specific rotation.

0.4 Tartaric acid is used as acidulant in soft drink, confectionery, bakery, gelation dessert industries, textile industry and silvering glass mirrors. It is also used in photography, tanning, ceramic industries, besides as raw material for various tartrates.'

[Page 3, Table 1, *Sl No.* (i), col 4 and 5] — Add '* (204 to 206)'.

[Page 3, Table 1, *Sl No.* (x)] — Insert a new requirement as follows:

| (1) | (2) | (3) | (4) | (5) | (6) |
|-----|---|-----|-----------------------------------|-----|-----------------------|
| xi) | †Optical rotation in 20 percent solution in water | ← | $+11.9^{\circ}$ to 12.3° | → | IS 326 (Part 4), 1980 |

(Page 3, Table 1) — Insert at the end of the table:

*This value is applicable to synthetic material only.

†This requirement shall be nil in the case of synthetic material.'

(PCD 9)

Reprography Unit, BIS, New Delhi, India

TABLE I REQUIREMENTS FOR TARTARIC ACID

(Clause 4.2)

| SL No. | CHARACTERISTIC | REQUIREMENTS FOR GRADE | | | METHOD OF TEST (REF TO CL No. OF APPENDIX B) |
|--------|--|---|---|--------------------|--|
| | | Technical | Pure | Analytical Reagent | |
| (1) | (2) | (3) | (4) | (5) | (6) |
| i) | Melting point | — | 169° to 171°C | 169° to 171°C | B-2 |
| ii) | Loss on drying, percent by weight, <i>max</i> | 2.5 | 1.0 | 1.0 | B-3 |
| iii) | Tartaric acid ($C_4H_4O_6$) content, percent by weight on dry weight, <i>min</i> | 98.0 | 99.5 | 99.5 | B-4 |
| iv) | Ash, percent by weight, <i>max</i> | — | 0.1 | 0.02 | B-5 |
| v) | Sulphates (as SO_4), percent by weight, <i>max</i> | 0.05 | 0.05 | 0.002 | B-6 |
| vi) | Heavy metals (as Pb), percent by weight, <i>max</i> | 0.001 | 0.001 | 0.000 8 | B-7 |
| vii) | Arsenic (as As_2O_3), parts per million, <i>max</i> | 18 | 1 | 1 | B-8 |
| viii) | Oxalate | — | To satisfy the requirements of the test | | B-9 |
| ix) | Copper and iron | To satisfy the requirements of the test | | | B-10 |
| x) | Chlorides (as Cl), percent by weight, <i>max</i> | — | — | 0.000 5 | B-11 |

APPENDIX A

(Clause 3.1)

SAMPLING OF TARTARIC ACID

A-1. GENERAL OF REQUIREMENTS SAMPLING

A-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

A-1.1 Samples shall not be taken at a place exposed to weather.

A-1.2 The sampling instrument shall be clean and dry when used.

A-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contaminations.

A-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

A-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers, on which the material has no action.

A-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

A-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

A-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

A-2. SCALE OF SAMPLING

A-2.1 Lot — All the containers in a single consignment of one grade of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

A-2.2 Gross Sample — A number of containers, not less than the sample size indicated in Table II, shall be selected at random from a lot for the purpose of drawing samples for test. These containers shall constitute the gross sample.

TABLE II MINIMUM NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING FROM VARIOUS SIZES OF LOTS

| LOT SIZE | SAMPLE SIZE |
|----------------|-------------|
| 2 to 8 | 2 |
| 9 .. 27 | 3 |
| 28 .. 64 | 4 |
| 65 .. 125 | 5 |
| 126 .. 216 | 6 |
| 217 .. 343 | 7 |
| 344 .. 512 | 8 |
| 513 .. 729 | 9 |
| 730 .. 1 000 | 10 |
| 1 001 .. 1 331 | 11 |

A-3. TEST SAMPLES AND REFEREE SAMPLE

A-3.1 Preparation — To prepare a set of test samples, draw with an appropriate sampling instrument, small portions of the material from different parts of each container in the gross sample selected for sampling and freshly opened. The sample prepared by mixing the portions from each container selected for sampling shall not exceed 250 g. Divide this composite sample into the required number of reduced samples.

A-3.2 Three sets of test samples, not less than 50 g each, representative of each container selected for sampling, shall be transferred immediately to thoroughly dried bottles which are sealed air-tight with glass stoppers. These shall be labelled with all the particulars of sampling given under A-1.7. One set of the test samples shall be sent to the purchaser and one to the vendor.

A-3.3 Referee Sample — The third set of the test samples, bearing the seals of the purchaser and the vendor, shall constitute the referee sample to be used in case of dispute between the purchaser

and the vendor. It shall be kept at a place as agreed between the purchaser and the vendor.

A-4. TESTS FOR ACCEPTANCE

A-4.1 Examination and Tests — The purchaser may examine and test each of the reduced samples constituting a test sample separately for compliance with the requirements of this standard; or he may prepare, for the purpose of such examination and at any stage of the progress of the examination, a composite sample representative of the whole lot, by mixing all the reduced samples constituting the test sample.

A-4.2 Criterion for Judgement — When the individual reduced samples in a test sample are separately examined and the results vary from one reduced sample to another so as to show that one or more results are outside the limits prescribed in the specification, the criterion for judging the quality of the lot for the purpose of acceptance on the basis of the results obtained shall be at the discretion of the purchaser, unless otherwise previously agreed between the purchaser and the vendor.

APPENDIX B

(Clause 4.2)

ANALYSIS OF TARTARIC ACID

B-1. QUALITY OF REAGENTS

B-1.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

B-2. DETERMINATION OF MELTING POINT

B-2.1 Apparatus — The apparatus shall consist of the following parts:

B-2.1.1 Glass Heating Vessel — of suitable construction and capacity.

B-2.1.2 Stirrer — capable of rapid mixing of the liquid.

B-2.1.3 Thermometer — The dimensions of the thermometer shall be as follows:

| | |
|---------------------|-------------------------------|
| Range | 85° to 215°C |
| Immersion | 100 mm |
| Stem diameter | 5.5 to 7 mm |
| Bulb length | 10 to 15 mm |
| Bulb diameter | Not greater than that of stem |
| Overall length, max | 310 mm |

Length of graduated portion, min 160 mm

Distance of lowest division to bottom of bulb 105 to 115 mm

B-2.1.4 Capillary Glass Tubes — made of soft glass, closed at one end, with a wall thickness of 0.10 to 0.15 mm, of sufficient length to allow the open end to be above the surface of the liquid in the heating tube, and of internal diameter of 0.8 to 1.2 mm.

B-2.1.4.1 The tubes should preferably be kept sealed at both ends and cut when required.

B-2.2 Procedure — Dry a small quantity of the finely powdered material in a vacuum desiccator. Transfer a portion of it to a dry capillary glass tube and pack the powder by tapping over hard surface so as to form a tightly packed column 2 to 4 mm in height. Heat a suitable liquid in the heating vessel and regulate, prior to the introduction of the capillary tube, the rate of rise of temperature to 3°C per minute, stirring constantly. When the temperature reaches 160°C, insert the capillary tube, attaching it to the thermometer so that the closed end is near the middle of the thermometer. Adjust the thermometer so that the immersion mark is at the level of the liquid. Note the temperature at which liquefaction of the material

occurs, as indicated by the formation of a definite meniscus. Regard this temperature as the melting point of the substance.

B-3. DETERMINATION OF LOSS ON DRYING

B-3.1 Procedure — Introduce about 5 g of the material in a tared weighing bottle provided with a glass stopper and weigh accurately. Remove the stopper and heat for three hours in a well ventilated air-oven maintained at $105^{\circ} \pm 2^{\circ}\text{C}$. Cool in a desiccator, replace the stopper and weigh. Repeat the process at intervals of one hour till constant weight is maintained. Reserve the dried material for test under B-4.

B-3.2 Calculation — Calculate the loss in weight as percentage of the weight of the material taken for the test.

B-4. DETERMINATION OF TARTARIC ACID ($\text{C}_4\text{H}_6\text{O}_6$) CONTENT

B-4.1 Reagents — The following reagents are required.

B-4.1.1 Standard Sodium Hydroxide Solution — approximately 1 N.

B-4.1.2 Phenolphthalein Indicator — Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit, 95 percent by volume (conforming to IS: 323-1952), that has previously been neutralized to the indicator.

B-4.2 Procedure — Dissolve about 3 g of the dried material (see B-3.1), accurately weighed, in 100 ml of water and titrate with standard sodium hydroxide solution using phenolphthalein as indicator.

B-4.3 Calculation

$$\text{Tartaric acid } (\text{C}_4\text{H}_6\text{O}_6) \text{ content, percent by weight} = \frac{7.505 \text{ } VN}{W}$$

Where

V = volume in ml of standard sodium hydroxide solution used up by the material,

N = normality of standard sodium hydroxide solution, and

W = weight in g of the material taken for the test.

B-5. DETERMINATION OF ASH

B-5.1 Procedure — Weigh accurately about 10 g of the material in a tared flat bottom silica dish of about 20 ml capacity. Ignite slowly, cool in a desiccator and weigh. Repeat the ignition till constant weight.

B-5.2 Calculation — Find the weight of the residue and express it as percentage of the weight of the material taken for the test.

B-6. TEST FOR SULPHATES (as SO_4)

B-6.1 Apparatus

B-6.1.1 Nessler Tubes — two flat bottomed tubes of thin, colourless glass, about 25 mm in diameter and about 150 mm in length, graduated at 50 ml. The depth measured internally from the graduated mark to the bottom shall not vary by more than 2 mm in the tubes used.

B-6.2 Reagents — The following reagents are required.

B-6.2.1 Dilute Hydrochloric Acid — approximately 4 N.

B-6.2.2 Barium Chloride Solution — Dissolve 12.2 g of barium chloride crystals in water and make up to 100 ml.

B-6.2.3 Standard Sulphuric Acid — 0.01 N.

B-6.3 Procedure for Technical and Pure Grades — Dissolve 2.5 g of the material in water and transfer it to a Nessler tube. Add 1 ml of dilute hydrochloric acid and 1 ml of barium chloride solution. Make up the solution to 50 ml. Carry out a control test, using 2.5 ml of standard sulphuric acid in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the turbidities produced in the two Nessler tubes five minutes after the addition of the barium chloride solution.

B-6.3.1 The limits prescribed in Table 1 for the technical and pure grades shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

B-6.4 Procedure for Analytical Reagent Grade — Dissolve 5.0 g of the material in 50 ml of water. Add 1 ml of dilute hydrochloric acid and 1 ml of barium chloride solution and allow to stand for one hour.

B-6.4.1 The limit prescribed in Table I shall be taken as not having been exceeded if the test with the material does not produce any turbidity or precipitate.

B-7. TEST FOR HEAVY METALS (as Pb)

B-7.1 Apparatus

B-7.1.1 Nessler Tubes — same as under B-6.1.1.

B-7.2 Reagents — The following reagents are required.

B-7.2.1 Ammonium Hydroxide Solution — sp-gr 0.90.

B-7.2.2 Standard Lead Solution — Dissolve 1.60 g of lead nitrate $[\text{Pb}(\text{NO}_3)_2]$ in water, add 1 ml of concentrated nitric acid (conforming to IS: 264-1950) and make up the volume to 1 000 ml mark. Transfer exactly 10 ml of the solution into a volumetric flask and dilute it again with water to 1 000 ml mark. One millilitre of this solution contains 0.01 mg of lead (Pb). The solution should be freshly prepared.

B-7.2.3 Dilute Acetic Acid — approximately 1 N.

B-7.2.4 Phenolphthalein Indicator — Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit, 95 percent by volume (conforming to IS: 323-1952), that has previously been neutralized to the indicator.

B-7.2.5 Hydrogen Sulphide — gas.

B-7.2.6 Glacial Acetic Acid — conforming to IS: 695-1955.

B-7.3 Procedure for Technical and Pure Grades — Dissolve 4.0 g of the material in 20 ml of water and add one drop of phenolphthalein indicator, followed by ammonium hydroxide solution until the solution is faintly pink. Transfer this solution to a Nessler tube, and add 4 ml of dilute acetic acid and make up the volume to 50 ml mark. To the other Nessler tube add 4 ml of dilute acetic acid and 4 ml of standard lead solution and make up the volume to 50 ml mark. Pass hydrogen sulphide through both the solutions for a few seconds and compare the colour in the two tubes.

B-7.3.1 The limits prescribed in Table I for the technical and pure grades shall be taken as not having been exceeded if the colour produced in the first tube is not deeper than that in the second tube.

B-7.4 Procedure for Analytical Reagent Grade — Dissolve 2.5 g of the material in 40 ml of water and add 10 ml of ammonium hydroxide solution. Transfer this solution to a Nessler tube. In the other Nessler tube, mix 2 ml of standard lead solution with 1 ml of glacial acetic acid. Add 10 ml of ammonium hydroxide solution and make up the volume to 50 ml mark. Pass hydrogen-sulphide through the solution for a few seconds.

B-7.4.1 The limit prescribed in Table I shall be taken as not having been exceeded if the intensity of colour produced in the first tube is not deeper than that produced in the second tube.

B-8. TEST FOR ARSENIC (as As_2O_3)

B-8.1 Apparatus — The apparatus consists of the following parts assembled as shown in Fig 1.

B-8.1.1 Gutzeit Bottle — wide mouthed, capacity 60 ml.

B-8.1.2 Narrower Tube — A, made of Pyrex or other equivalent glass, 100×4 mm, with a constriction at a distance of 50 mm from the upper end.

B-8.1.3 Wider Tube — B, also made of Pyrex or other equivalent glass, 120×12.5 mm, with a constriction at a distance of 50 mm from the upper end.

B-8.1.4 Rubber Stoppers — two, one to fit the Gutzeit bottle and bored with a hole to take the wider tube B, and the other to fit inside the wider tube and bored with one hole to take the narrower tube A.

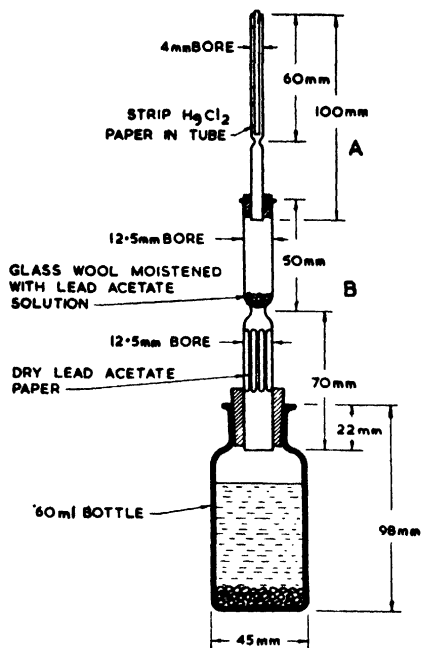


FIG 1 APPARATUS FOR DETERMINATION OF ARSENIC

B-8.2 Reagents

B-8.2.0 Blanks should be run on all reagents used for this work. The reagents are considered arsenic-free if no stain is produced on mercuric chloride paper after 45 minutes. The following reagents are required.

B-8.2.1 Sodium Hydroxide Solution — approximately 20 percent w/v, arsenic-free.

B-8.2.2 Dilute Sulphuric Acid — approximately 5 N, arsenic-free.

B-8.2.3 Concentrated Sulphuric Acid — sp-gr 1.84 (conforming to IS: 266-1950).

B-8.2.4 Standard Arsenic Trioxide Solution — Dissolve 1.00 g of re-sublimed arsenic trioxide (As_2O_3) in 25 ml of sodium hydroxide solution and neutralize with dilute sulphuric acid. Dilute with freshly distilled water, to which 10 ml of concentrated sulphuric acid per litre has been added, and make up to 1 000 ml. Again dilute 10 ml of this solution to 1 000 ml with water containing sulphuric acid, and finally dilute 100 ml of the latter solution to 1 000 ml with water containing sulphuric acid. One millilitre of the final solution contains 0.001 mg of arsenic trioxide (As_2O_3).

B-8.2.5 Standard Stains — Make the standard stains on sensitized mercuric chloride paper (see B-8.2.9) with the appropriate quantities of standard arsenic trioxide solution. Place the requisite

amount of the standard arsenic trioxide solution in the Gutzeit bottle with the amounts of reagents prescribed for the regular tests and the run made exactly as prescribed for the regular procedure under B-8.3.

B-8.2.5.1 Preservation of stains — Dip the strips of the sensitized paper with the arsenic stain in molten paraffin (free from water) and mount on a sheet of white paper, folded back to form a cylinder. Place the tube in a glass test-tube containing phosphorus pentoxide, which is then closed by a stopper. Keep the stained strips dry, otherwise the stain soon fades, hence perfectly dry the glass test-tube and the paper on which the strips are mounted. While not in use, keep the strips in an empty hydrometer case, as light will gradually fade the colour. Fresh stains should be prepared periodically, and a stain suspected to have faded shall not be used. Fresh stains shall be prepared in case of dispute.

B-8.2.6 Lead Acetate Solution — Prepare one percent solution of lead acetate [$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$] with sufficient acetic acid added to clear the solution.

B-8.2.7 Dry Lead Acetate Test Paper for Removal of Hydrogen Sulphide — Soak large sheets of qualitative filter paper in lead acetate solution and dry. Cut the paper into pieces measuring 70×50 mm.

B-8.2.8 Mercuric Chloride Solution — approximately 0.35 percent (w/v).

B-8.2.9 Sensitized Mercuric Chloride Paper — Cut 50×50 cm Swedish Filter Paper No. 0 into four equal squares. Dip the strips into mercuric chloride solution. The paper should be of uniform thickness, otherwise there may be an irregularity in the length of the stain for the same amount of arsenic. Hang the paper and dry it in air, free from gas fumes, hydrogen sulphide being particularly undesirable. When dry, trim off about 1 cm of the outer edge and cut the paper into strips, $12 \text{ cm} \times 2.5 \text{ mm}$. Preserve the papers in bottles with tight-fitting stoppers. Paper with a white deposit of mercuric chloride shall not be used. Stains shall be made with each batch of paper.

B-8.2.10 Mixed Acid — Dilute one volume of concentrated sulphuric acid with four volumes of water, and to this add 10 g of sodium chloride for each 100 ml of solution.

B-8.2.11 Ferric Ammonium Sulphate Solution — Dissolve 84 g of ferric ammonium sulphate in water with 10 ml of mixed acid and make up to a litre.

B-8.2.12 Concentrated Hydrochloric Acid — sp-gr 1.16 (conforming to IS: 265-1950).

B-8.2.13 Stannous Chloride Solution — Dissolve 80 g of stannous chloride in 100 ml of water containing 5 ml of concentrated hydrochloric acid.

B-8.2.14 Zinc — Treat arsenic-free zinc shots passing through Indian Standard Sieve 570 and retained on Indian Standard Sieve 280 (see Appendix C), with hydrochloric acid, until the surface of the zinc becomes clean and dull. Wash and

keep under water in a casserole, covered with a clock-glass to keep out dust.

B-8.3 Procedure — Place the dry lead acetate paper in the lower portion of the tube B, and glass wool moistened with lead acetate solution in its upper portion. Place the strip of sensitized mercuric chloride paper in tube A, and connect the tubes together with a rubber stopper. Introduce 20 ml of water into the Gutzeit bottle and add 5 ml of concentrated sulphuric acid. Add 2 ml of ferric ammonium sulphate solution and 0.5 ml of stannous chloride solution. Dissolve 1.00 g of the material in 20 ml of water and transfer the solution completely into the Gutzeit bottle, washing the dish with minimum quantity of water. Make up the volume to 50 ml, mix the contents and drop about 10 g of zinc shots. Immediately fit in position the rubber stopper carrying the tube B. Place the bottle in a warm place at about 40°C . At the end of 45 minutes, remove the strip of sensitized mercuric chloride paper by means of tweezers and compare the stain with the standard stain produced with 0.001 mg of arsenic trioxide (As_2O_3) in the case of analytical reagent and pure grades, and 0.018 mg of arsenic trioxide in the case of technical grade.

B-8.3.1 The limits prescribed in Table I shall be taken as not having been exceeded if the length and intensity of the stain produced in the test with the material is not greater and deeper than that produced with the specified quantities (see B-8.3) of standard arsenic trioxide solution.

B-9. TEST FOR OXALATE

B-9.1 Reagents — The following reagents are required.

B-9.1.1 Ammonium Hydroxide Solution — sp-gr 0.90.

B-9.1.2 Calcium Sulphate Solution — saturated solution in water.

B-9.2 Procedure — Dissolve 1.0 g of the material in water and make up the volume to 10 ml. Neutralize the solution with ammonium hydroxide solution and add 10 ml of calcium sulphate solution.

B-9.2.1 The material shall be regarded to have satisfied the requirement of the test if no turbidity is produced.

B-10. TEST FOR COPPER AND IRON

B-10.1 Apparatus

B-10.1.1 Nessler Tubes — same as under B-6.1.1.

B-10.2 Reagents — The following reagents are required.

B-10.2.1 Ammonium Hydroxide Solution — sp-gr 0.90.

B-10.2.2 Sodium Sulphide Solution — approximately 10 percent.

B-10.2.3 Potassium Cyanide Solution — lead-free. Dissolve 10 g of potassium cyanide in 90 ml

of water, add 2 ml of hydrogen peroxide solution, allow to stand for 24 hours and make up to 100 ml with water.

B-10.3 Procedure — Dissolve 2 g of the material for analytical reagent and pure grades and 3 g in case of technical grade in 40 ml of water. Add 10 ml of ammonium hydroxide solution and 5 drops of sodium sulphide solution. Carry out a control test in the other Nessler tube, using the same quantities of the material and the reagents in the same total volume of the reaction mixture and containing, besides, 1 ml of potassium cyanide solution. Compare the colours of the solutions in both the tubes.

B-10.3.1 The material shall be regarded to have satisfied the requirements of the test if the intensity of colour produced in the first tube is at the most

only slightly deeper than that produced in the control test.

B-11. TEST FOR CHLORIDES (as Cl)

B-11.1 Reagents — The following reagents are required.

B-11.1.1 Dilute Nitric Acid — approximately 4 N.

B-11.1.2 Silver Nitrate Solution — approximately 4 percent (w/v).

B-11.2 Procedure — Weigh 2.0 g of the material and dissolve in 50 ml of water. Add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution.

B-11.2.1 The limit prescribed in Table I shall be taken as not having been exceeded if the test with the material does not show any opalescence within 5 minutes.

APPENDIX C

(Clauses 0.4 and B-8.2.14)

SIEVE SIZES

C-1. COMPARATIVE SIEVE DESIGNATIONS

C-1.1 Table III gives comparative designations

of BS and ASTM Test Sieves against the corresponding IS Sieve designations. Only the sizes quoted in the standard are included in Table III.

TABLE III COMPARATIVE DESIGNATIONS OF IS, BS AND ASTM TEST SIEVES

| Sl. No. | IS* | | BS | | ASTM | |
|---------|-------------------|--------------------|-------------------|--------------------|-------------------|--------------------|
| | Sieve Designation | Aperture (Microns) | Sieve Designation | Aperture (Microns) | Sieve Designation | Aperture (Microns) |
| i) | 570 | 5 660 | — | — | 3½ | 5 660 |
| ii) | 280 | 2 818 | 6 | 2 812 | 7 | 2 830 |

*See also IS: 460-1953